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THE TALIANI TEST AS A CRITERION OF PROPELLANT STABILITY

By

Carl Boyars
W. G. Gough

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U.S. NAVAL POWDER FACTORY

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FOREWORD

This investigation was performed under Bureau of Ordnance Task Assignment NPF-13-Re2d-02-6-52. Memorandum Reports No. 12 and 20, issued on 15 October 1951 and 15 May 1952, served as progress reports on the early phases of this work.

This report has been reviewed for technical accuracy by R. G. Parnell and W. J. Moore and was submitted for publication 9 December 1952. This work was performed while Mr. F. C. Thames was Director of Research and Development.

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Head, Chemical Physics
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ACKNOWLEDGEMENT

M. E. Baicar was responsible for preparation of deteriorated samples used. Mrs. C. J. Wright and C. V. Jansen assisted in carrying out the tests. Modified N-4 propellants were prepared by the Engineering Division of this Department.

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ABSTRACT

An investigation of the utility of Taliani tests for supplying information about the stability of double-base propellants ~~has been~~ was carried out. A definite correlation between the degree of deterioration of a propellant and its behavior in a Taliani test under oxygen ~~has been~~ was found.

INTRODUCTION

The Taliani test is essentially one in which a sample of propellant is heated in a constant volume system, starting with a fixed pressure of gas (usually one atmosphere) and measuring the rate of pressure change. The original apparatus (1,2) has been modified greatly by workers at the California Institute of Technology[3] and further improved at the Naval Powder Factory[4]. In studies of the stabilization of double base powder carried out at the Allegany Ballistics Laboratory[5], the conclusion was reached that "Taliani data obtained under nitrogen represents a good test for gas bubble formation for evaluation of rocket propellants, and the test conducted under air or oxygen gives no information other than the rate of oxygen consumption."

The problem of propellant stability in general can be divided into two parts: (1) the length of time the propellant can be stored before depletion of stabilizer and subsequent accelerated decomposition results in spontaneous ignition or deterioration of the propellant (chemical safe-life); and (2) the length of storage it can undergo before a significant change in ballistic properties occurs due to a breakdown of physical structure (physical safe-life). The term "safe-life" used subsequently in this report refers to "chemical safe-life". Spurlin[6] has discussed the different types of storage failure in some detail, and the Naval Powder Factory has reported investigations of the problems of storage of JP and JPN ballistites[7].

The degree of deterioration of a stored propellant can be estimated by different methods such as chemical determination of stabilizer content (as compared to the original) or determination of the changes in viscosity of the nitrocellulose or changes in mechanical properties of the propellant. Unfortunately, such data is often not easily obtained and is difficult to interpret in terms of residual safe-life properties of the propellant. A good stability test that would give a precise measure of degree of deterioration is desirable, and this, together with the need for more information about the phenomena of degradation of propellants, inspired this investigation.

EXPERIMENTAL PROCEDURE

In the initial phase of this investigation, 45-g JPN surveillance samples which had been stored at 65.5°C for varying periods were selected to give a wide range of residual safe-life. Subsequently, aged samples were prepared by placing 100-g samples of sheet N-4 (Lot PAE. 133) in surveillance bottles in an oven at 80°C. The samples were put in at weekly intervals until the first one fumed. At this point all were removed, providing a series of samples of different residual safe-life. A separate series of PAE 133 aged at 80°C was prepared for Taliani tests at 110°C and 120°C, respectively. The time-to-fumes at 80°C was 93 days in one case and 105 days in the other. This difference is probably due to slight differences in oven temperature. A series of sheet JPN (Lot IXR-37) samples was prepared similarly. Other groups of sheet N-4 (Lot PAE 133) samples were placed in a 65.5°C surveillance magazine and withdrawn at 40-day intervals for testing. A series of modified N-4 (Lot IXR-47) propellants, differing from each other only in the stabilizer added, was prepared and subjected to Taliani tests.

The Taliani test apparatus and procedure have been described in Technical Report No. 25, pp. 34-35[4]. The propellant samples were ground in a Wiley cutting mill; portions which passed a U. S. 18 and were held on a U. S. 50 sieve were selected. The ground samples were weighed into Taliani test tubes. For the tests conducted under air, the samples were preheated in the bath for 30 minutes, after which the pressure was reduced to atmospheric and the initial reading taken. The tests under oxygen and nitrogen had samples preheated under air in the same manner, but, following the preheating period, the system was evacuated and flushed four times with the appropriate gas before the final addition of gas which brought the internal pressure above atmospheric. The pressure was then reduced to atmospheric by rapidly opening and closing a stopcock.

DISCUSSION

The Taliani test data are plotted in Figures 1 through 17 in the Appendix. Duplicate readings were taken each half hour and their averages plotted. Figures 1 and 12 show quite clearly that 110°C Taliani tests under nitrogen are of no value in detecting aging of JPN. Figures 6 and 9 lead to the same conclusion for N-4. Figure 2 indicates the usefulness of the 110°C test under oxygen for measuring degree of deterioration; Figures 4, 7, and 10 show even more clearly that this test correlates well with the condition of the powder if the time to reach a fixed positive pressure, e.g. 100 mm, is measured. Figures 13 and 14 show that carrying out the oxygen Taliani test at 120°C provides a more rapid measurement without sacrificing the good correlation of the lower temperature test. Figures 3, 5, 8 and 11 show that a Taliani test under air reveals deterioration only during the later stages of the powder's safe-life.

These facts can be readily explained. One of the gases evolved in the Taliani test is NO. Under nitrogen, this gas does not contribute further to the decomposition process during the test period. Under air or oxygen, NO reacts to form NO_2, which then reacts with the powder. Thus, the immediate net result is a decrease in amount of gas present in the system, so long as effective quantities of stabilizer are present to pick up the NO_2. When the stabilizer becomes depleted, the NO_2 attacks the nitrate esters primarily and increases the decomposition rate of the powder.

Allowing for the volume occupied by sample, there are initially 6.7 ml of gas at 110°C and 1 atm pressure in the Taliani apparatus. This is equivalent to 2.13×10^{-4} moles. When the gas is oxygen, 4.26×10^{-4} moles of NO can be converted to NO_2. If the test is carried out in an atmosphere of air, 0.89×10^{-4} moles of NO can be oxidized. The number of moles of stabilizer in 1 g of a 1% ethyl centralite powder (JPN) is only 0.37×10^{-4}. There are 0.93×10^{-4} moles of stabilizer in 1 g of a 2% 2-nitrodiphenylamine powder (N-4). The amount of NO_2 which each molecule of stabilizer can absorb before becoming ineffectual has not been established. However, it is probable that, in a Taliani test under oxygen, centralite- or 2-nitrodiphenylamine-stabilized propellants containing no more than 2% stabilizer are completely converted to an unstable form through nitration of stabilizer. This would not hold true under air unless the powder had already been aged substantially.

The test under oxygen, in which the time required for a fixed pressure above atmospheric to be achieved is determined, is better able to distinguish between propellants in the early stages of deterioration because this time is dependent on the exact amount of effective stabilizer initially present. It is likely that the more rapid pressure increase shown in testing of propellants in the later stages of deterioration under air, as compared to oxygen, is due to early depletion of the oxygen by reaction with NO along with stabilizer depletion. After this occurs, all NO produced increases the total pressure. The increased pressure probably also accelerates the decomposition catalyzed by acidic products. Under oxygen, the NO produced must use up much more oxygen before it can increase the pressure directly. In the late stages of decomposition, NO_2 may be liberated from the powder itself.

Figures 15 through 17 show the Taliani behavior of a series of modified N-4 propellants. IXR-47A contains the customary 2% 2-nitrodiphenylamine. In IXR-47B, this stabilizer is replaced by the same percentage of carbazole; in IXR-47C, by diphenylamine; in IXR-47D, by ethyl centralite; and in IXR-47E, by a mixture of 1% 2-nitrodiphenylamine with 1% ethyl centralite. IXR-47F contains no stabilizer. To insure that no differences would occur in composition other than stabilizer, a single 150-lb batch of unstabilized N-4 was prepared and then divided into six parts. The appropriate stabilizer was then added by dry mixing.

As the graphs show, the test under nitrogen indicates the unstabilized propellant to be the most stable one and the propellant stabilized with the 1:1 mixture of 2-nitrodiphenylamine and centralite to be the least stable. This is, of course, contrary to all that is known about the relative safe-life of stabilized and unstabilized propellants. The test under oxygen gives results which fit the facts of safe-life much better. Here the unstabilized propellant reaches 100 mm pressure in under 6 hours. At 11 hours, the diphenylamine-stabilized propellant is the next to fail. Diphenylamine is notorious as a poor stabilizer for double-base propellants because of its alkalinity. The other four propellants all reach 100 mm pressure at about the same time, approximately 20 hours, the one stabilized with the centralite - 2-nitrodiphenylamine mixture lasting slightly longer than the others. Centralite and 2-nitrodiphenylamine, which are in three of these latter four propellants, are known to be good stabilizers for double-base propellants. According to Davis(8), carbazole is an excellent stabilizer at 110°C (the temperature of this test), but an extremely poor one at 60° and 75°C.* Naturally,

*The stabilizing power of carbazole and the other stabilizers in the modified N-4 compositions is now under investigation by measuring time-to-fumes at various temperatures.

any high temperature test is subject to errors involved in extrapolation of data and conclusions to lower temperatures at which different reactions may predominate.

Figures 18 through 21 illustrate how oxygen Taliani data can be used in the determination of residual safe-life of a propellant which has been in storage. The relationships between data obtained from the Taliani tests (the time required to achieve a pressure of +100 mm) and the percent of safe-life remaining have been plotted. The latter value has been computed from the ratio between length of storage at 80°C and time required for the sample to evolve NO_2 fumes at that temperature. Of course, estimates of the safe-life time of a new propellant formulation are best obtained by extrapolation of temperature-decomposition rate data to lower temperatures. In each case, the plot of the logarithm of the time required to reach 100 mm pressure vs percent of safe-life remaining approximates a straight line, and the least squares line is shown in each of the figures.

Some speculation on the theoretical interpretation of this data is in order here. It can reasonably be assumed that, for any propellant formulation, the time required to reach 100 mm pressure is equal to some constant value plus a factor proportional to the concentration of "active" stabilizer.

Thus: $$t_p = c + gs \qquad (1)$$

where t_p is the time required to reach 100 mm pressure in the oxygen Taliani test, c is a constant equal theoretically to the time required for the propellant formulation without stabilizer to reach 100 mm pressure, g is a proportionality constant, and s is the concentration of "active" stabilizer, a value which is a measure of the amount of NO_2 which can be picked up before the stabilizer becomes depleted.

$$s = \frac{t_p - c}{g} \tag{2}$$

$$\log s = \log(t_p - c) - \log g \tag{3}$$

The theoretical importance of these equations is based on the fact that, if equation (1) holds true, c can be evaluated by actual determination of the time for an unstabilized propellant to reach 100 mm pressure or by extrapolation of the oxygen Taliani data obtained periodically on a propellant being aged to the end of its safe-life. The values for t_p and c could be used in determining the pseudo-order of the stabilizer depletion reaction. If the reaction were zero-order, then

$$s = -kt + d \tag{4}$$

where t is the time of storage of the propellant at a fixed temperature, and d is a constant.

$$\frac{t_p - c}{g} = -kt + d \tag{5}$$

$$t_p - c = -gkt + gd = k't + d' \tag{6}$$

Thus, if the plot of (t_p-c) versus t yielded a straight line, the reaction would be shown to be pseudo-zero-order.

Similarly, if the reaction were first-order, then

$$-\log s=\left(\frac{k}{2.303}\right)t+d \qquad (7)$$

$$-\log(t_p-c)=\left(\frac{k}{2.303}\right)t+d-\log g=\left(\frac{k}{2.303}\right)t+d' \qquad (8)$$

If the plot of log (t_p-c) versus t yielded a straight line, the reaction would be pseudo-first-order and k, the specific reaction rate constant, could be evaluated from the slope.

It should be pointed out that the inhomogeneity of the propellant colloid restricts the precision with which quantitative measurements of the rate of loss of safe-life can be made. Consequently, a proper evaluation of c from Taliani data on propellants being decomposed in constant temperature storage would require many oxygen Taliani tests on many propellant lots of identical formulations so that statistical treatment could smooth out individual irregularities. The precision of the oxygen Taliani test as a measure of the residual safe-life of propellants could also be increased by thus obtaining additional data. The lower temperature Taliani tests permit greater precision than the high temperature ones by lessening the effect of slight variations in preheat time which are due to the manipulations involved in flushing the system.

The utility of the oxygen Taliani test for measuring the degree of propellant deterioration during storage has been demonstrated above, and the possible utility of such a test for evaluating proposed stabilizers has been indicated. The use of the test in specifications or as a control for the manufacture of propellants should be considered. The discrepancy between test time for the N-4 sheet lot PAE 133, a regular Picatinny Arsenal production lot, and the N-4 lot IIR-47A, prepared by dry addition of stabilizer to the unstabilized sheet, may be due to the differences in processing or to possible differences in composition. With suitable adjustment of sample size or of volume of the apparatus, the test can be applied to any solventless propellant based on nitrate esters by providing sufficient oxygen for complete depletion of stabilizer. Of course if gases other than NO are evolved rapidly, any fixed positive pressure may be achieved prior to exhaustion of stabilizer.

The nitrogen Taliani test is useful for quickly comparing rates of gas evolution from different propellants, but its validity as an absolute measure of the tendency of propellants to fissure is open to question. The permeability of different propellant compositions may be expected to vary as well as their gas evolution rates. Ernsberger and Olsen[9] have concluded, on the basis of their experimental work on evolution and diffusion of gases in ballistite, that the pressure built up in a solid 2-inch-diameter cylinder of JPN after storage in air for an extended period at 60°C may be insufficient to cause cracking.

An investigation of the effect on the oxygen Taliani test of incorporation of varying quantities of stabilizer into the nitrocellulose-nitroglycerin system is contemplated. This would provide further information about the validity of the assumption relating test time to "active" stabilizer content. The effect of varying the nitrocellulose-nitroglycerin ratio is also to be investigated.

SUMMARY

A Taliani test under oxygen has been found to give a quantitative measure of the residual safe-life of propellants in storage. A relationship between the amount of "active" stabilizer and the test result has been proposed. The oxygen Taliani test is also useful for evaluating proposed new stabilizers.

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F. M. Ernsberger and A. L. Olsen, Navord Report No. 1184, Part 3, NOTS 381, p. 13, April 1951. CONFIDENTIAL

APPENDIX

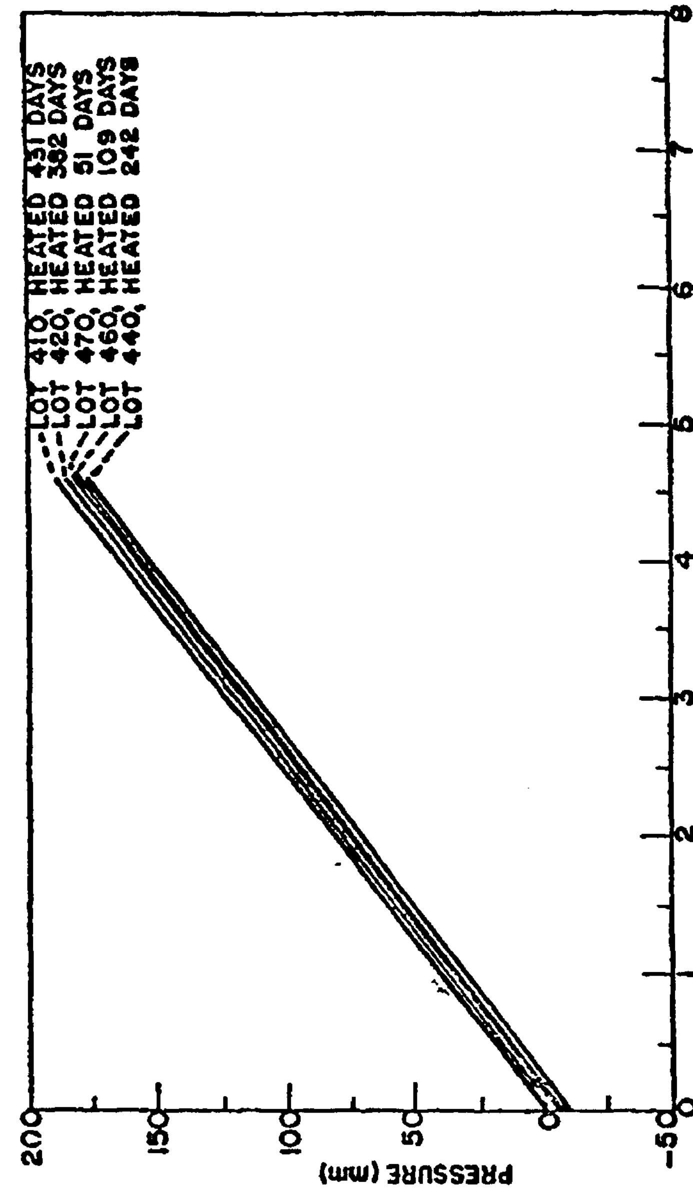

FIGURE 1. 110°C TALIANI TEST UNDER NITROGEN ON JPN SURVEILLANCE SAMPLES STORED AT 65.5°C.

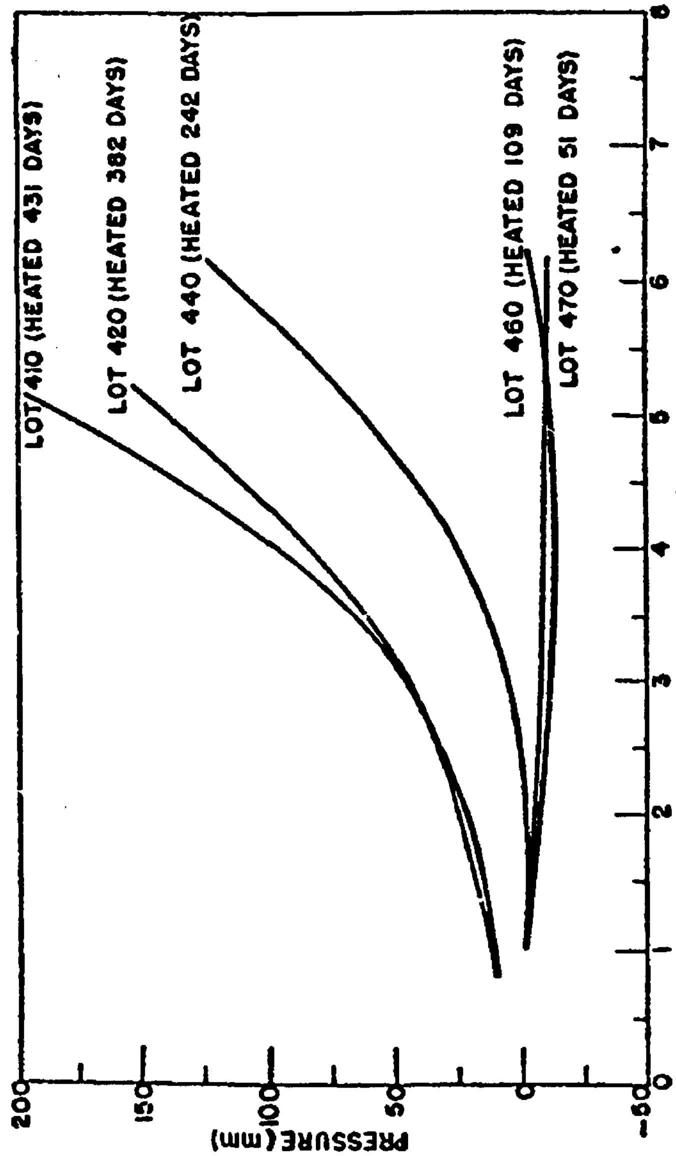

FIGURE 2. 110°C TALIANI TEST UNDER OXYGEN ON JPN SURVEILLANCE SAMPLES STORED AT 65.5°C.

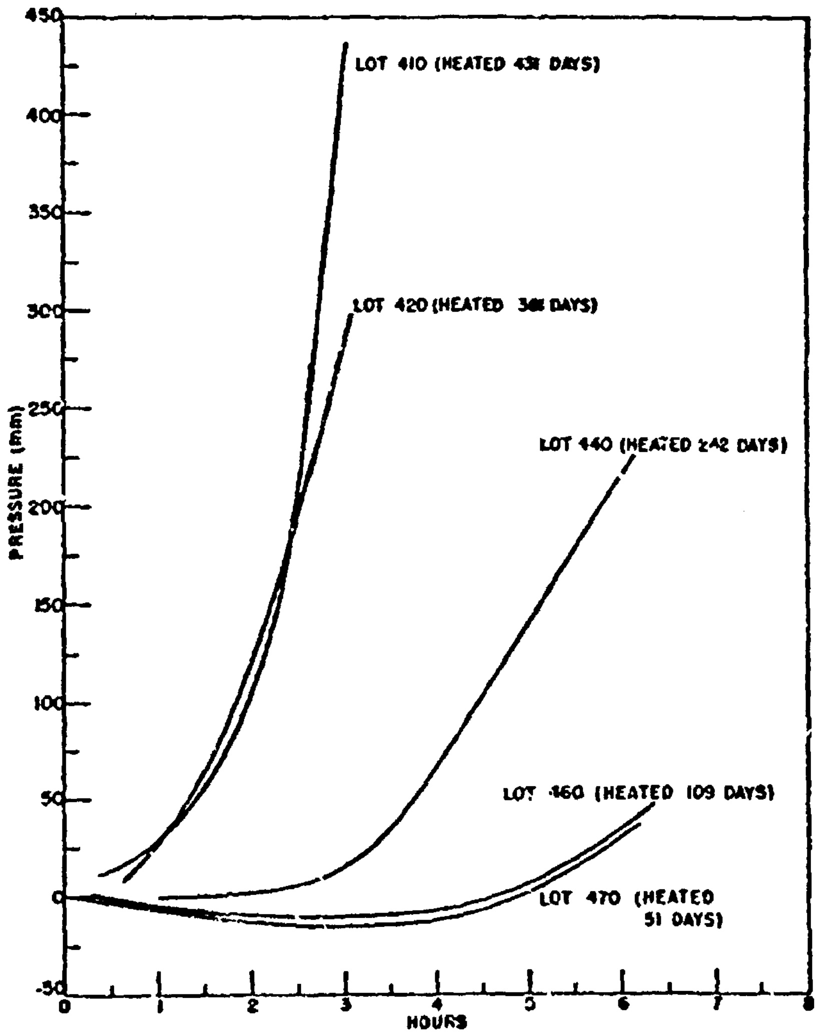

FIGURE 3. 110°C TALIANI TEST UNDER AIR ON JPN SURVEILLANCE SAMPLES STORED AT 65.5°C.

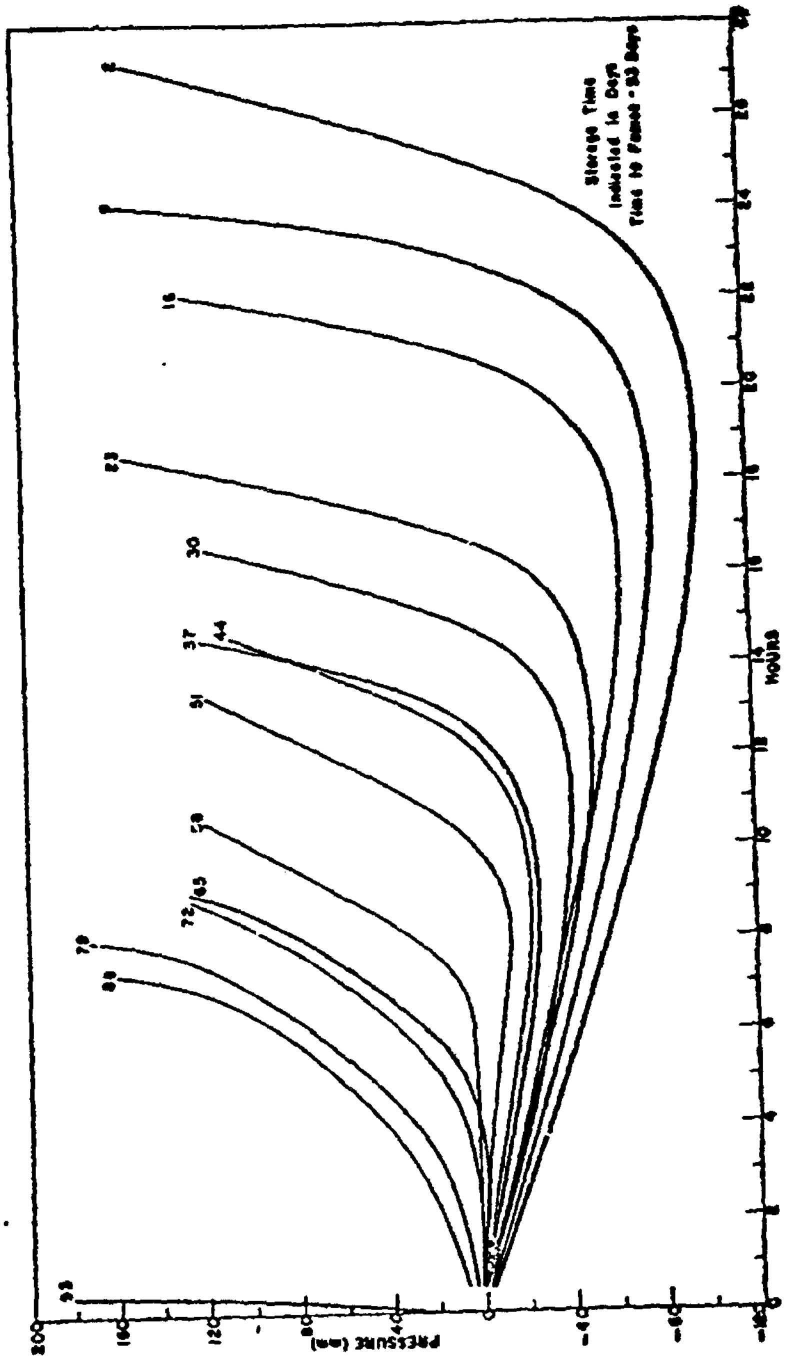

FIGURE 4. 110°C TALIANI TEST UNDER OXYGEN ON N-4 SAMPLES STORED AT 80°C.

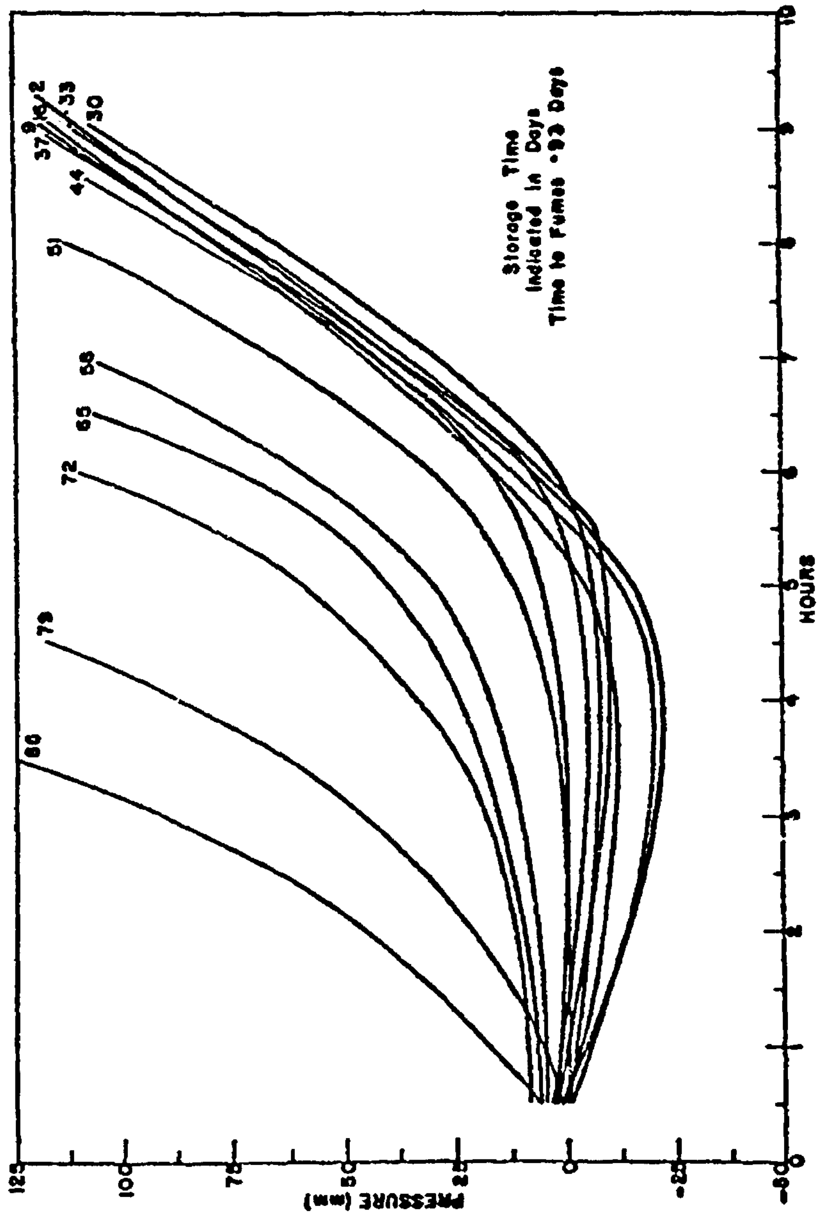

FIGURE 5. 110°C TALIANI TEST UNDER AIR ON N-4 SAMPLES STORED AT 80°C.

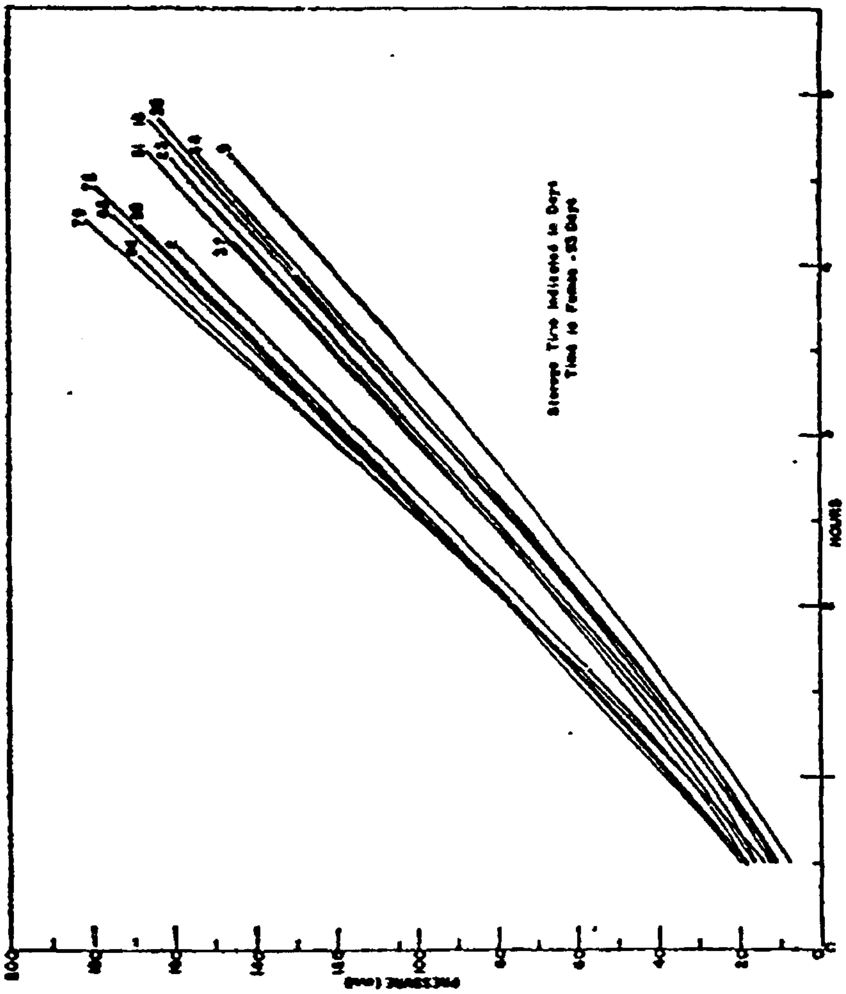

FIGURE 6. 110°C TALIANI TEST UNDER NITROGEN ON N-4 SAMPLES STORED AT 80°C.

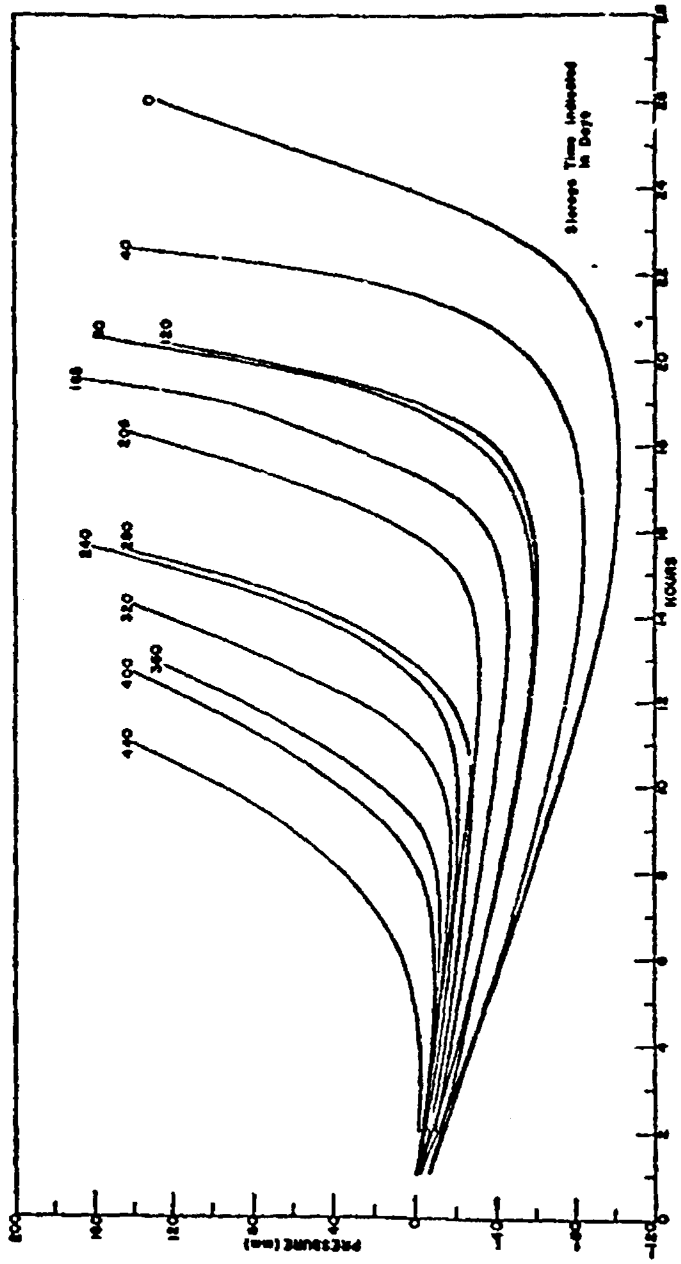

FIGURE 7. 110°C TALIANI TEST UNDER OXYGEN ON N-4 SAMPLES STORED AT 65.5°C

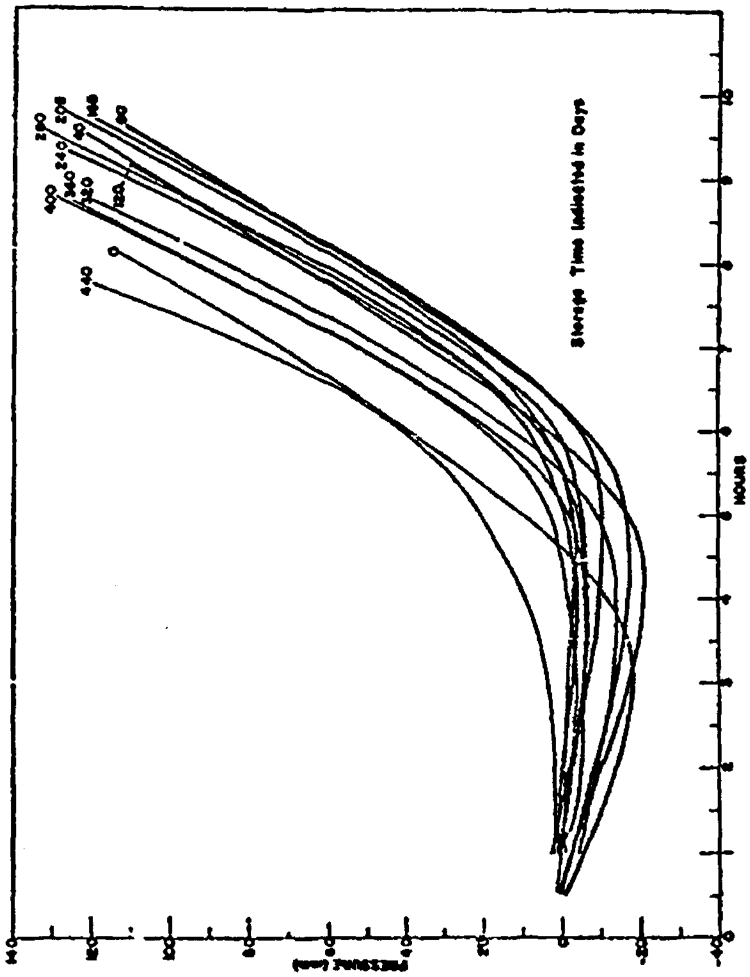

FIGURE 8. 110°C TALIANI TEST UNDER AIR ON N-4 SAMPLES STORED AT 65.5°C.

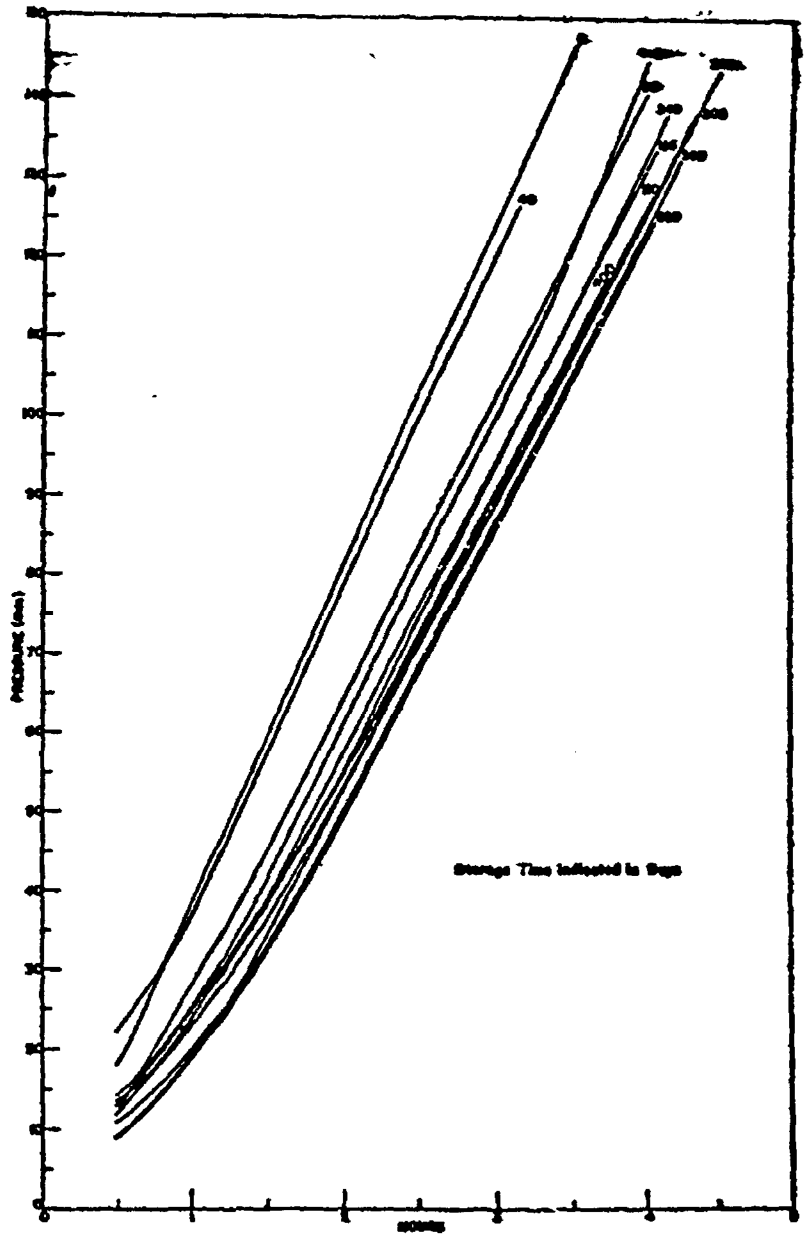

FIGURE 9. 110°C TALIANI TEST UNDER NITROGEN ON N-4 SAMPLES STORED AT 65.5°C.

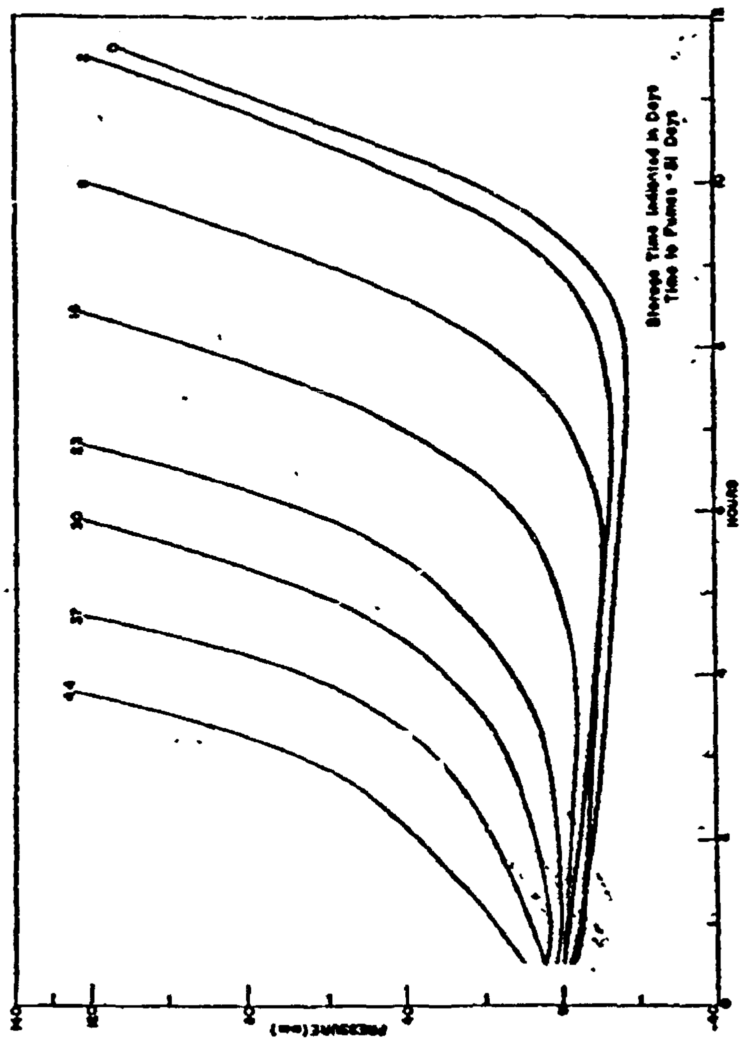

FIGURE 10. 110°C TALIANI TEST UNDER OXYGEN ON JPN SAMPLES STORED AT 80°C.

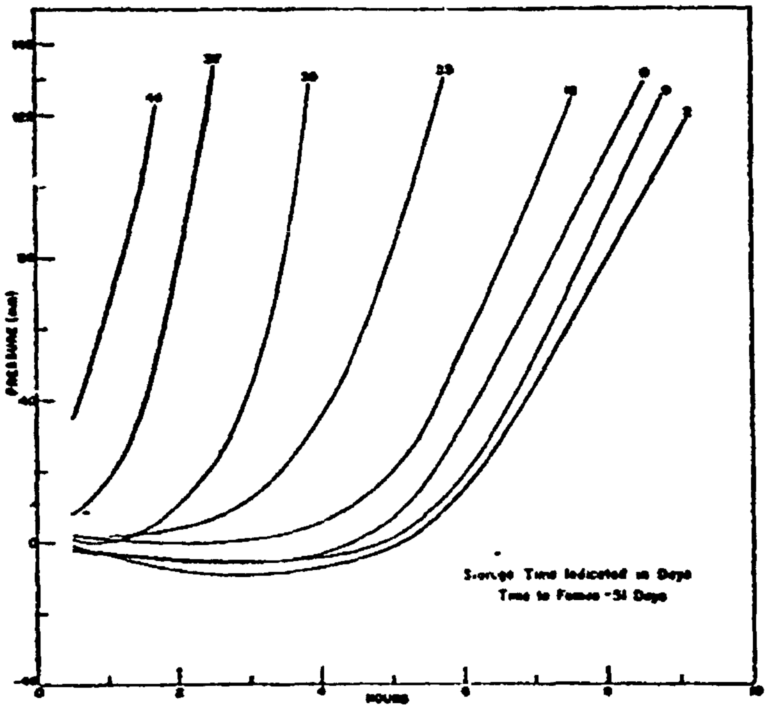

FIGURE 11. 110°C TALIANI TEST UNDER AIR ON JPN SAMPLES STORED AT 80°C.

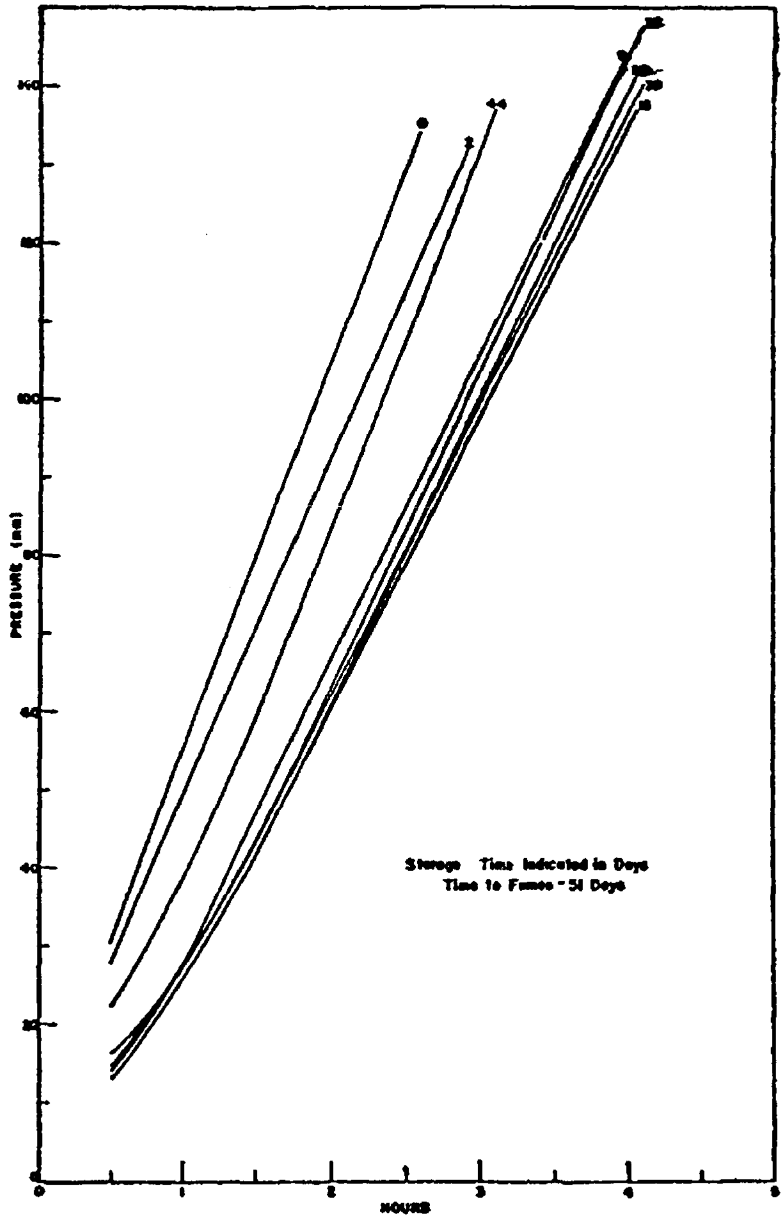

FIGURE 12. 110°C TALIANI TEST UNDER NITROGEN ON JP-4 SAMPLES STORED AT 80°C.

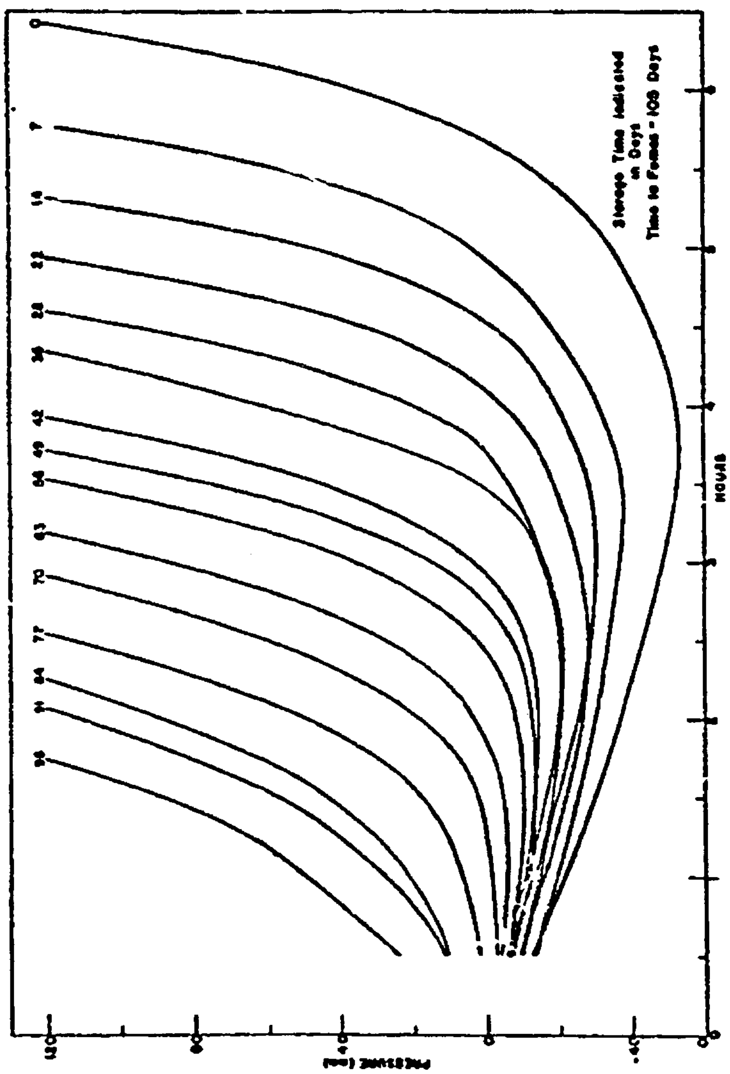

FIGURE 13. · 120°C TALIANI TEST UNDER OXYGEN ON N-4 SAMPLES STORED AT 80°C.

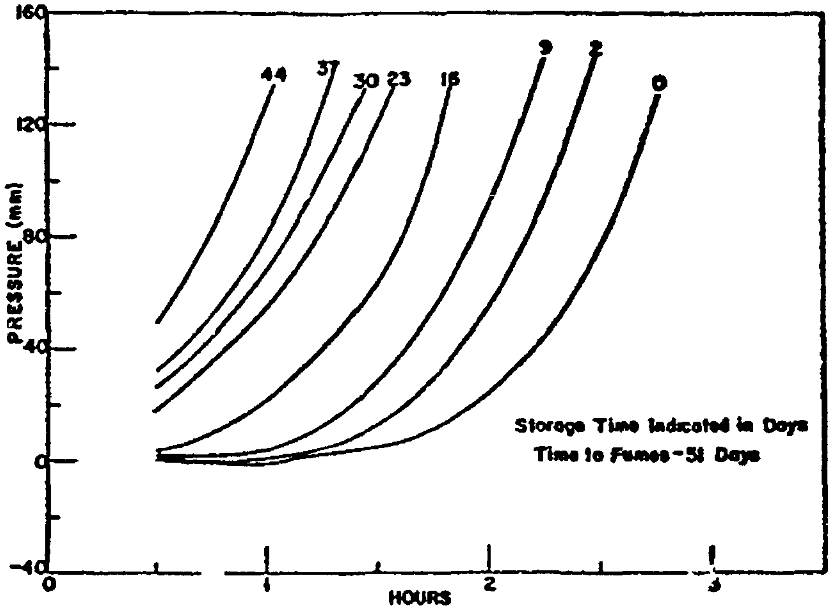

FIGURE 14. 120°C TALIANI TEST UNDER OXYGEN ON JPN SAMPLES STORED AT 80°C.

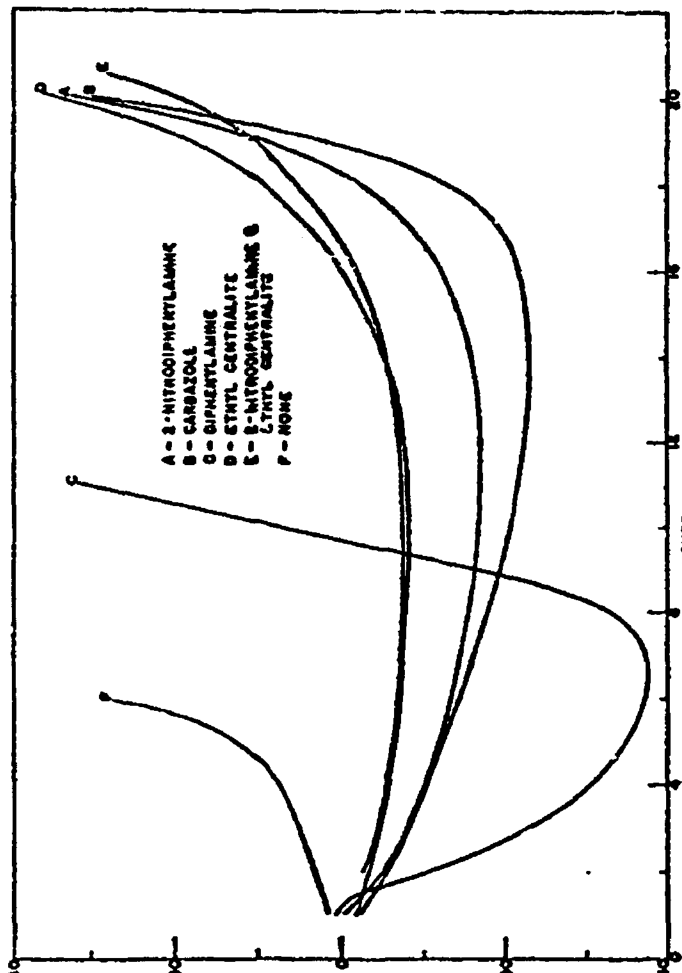

FIGURE 15. 110°C TALIANI TEST UNDER OXYGEN ON MODIFIED N-4 FORMULATIONS.

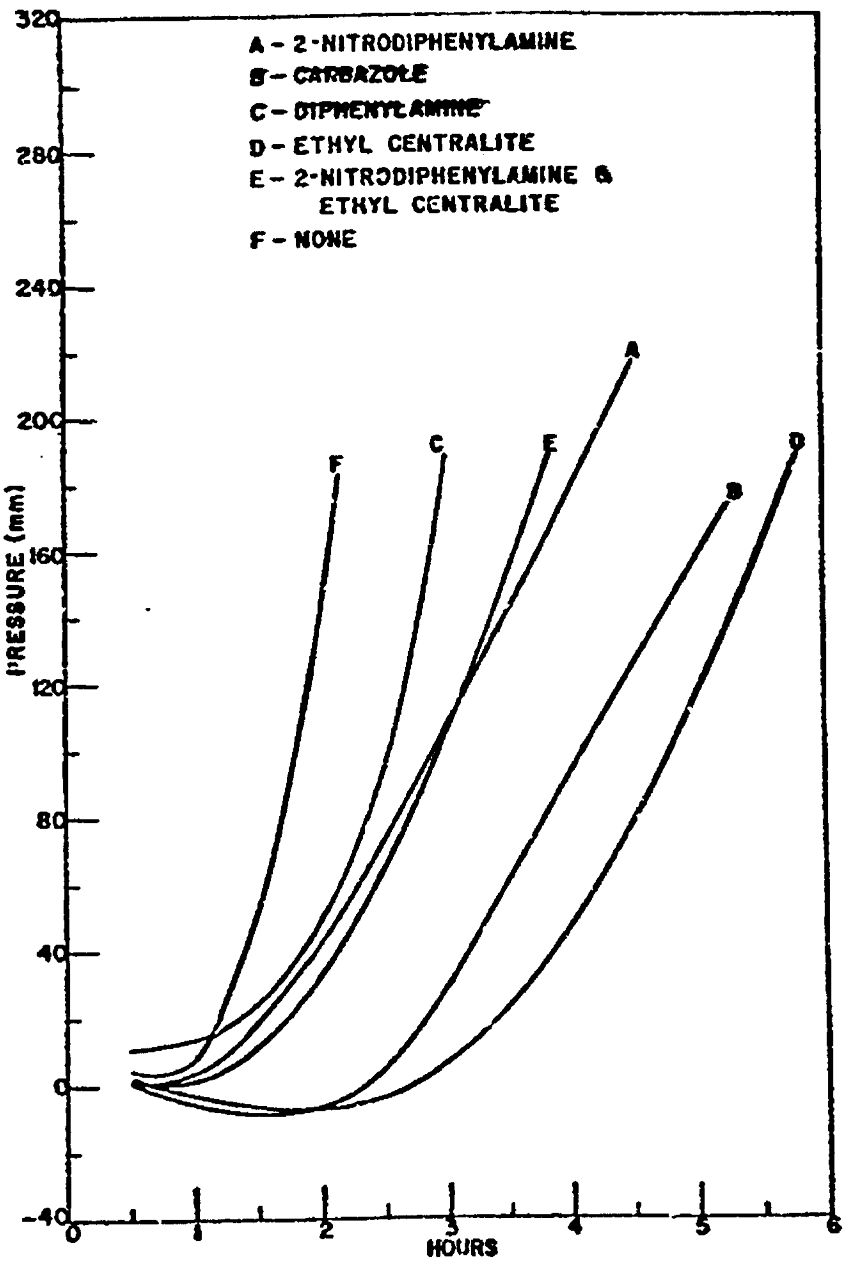

FIGURE 16. 110°C TALIANI TEST UNDER AIR ON MODIFIED N-4 FORMULATIONS

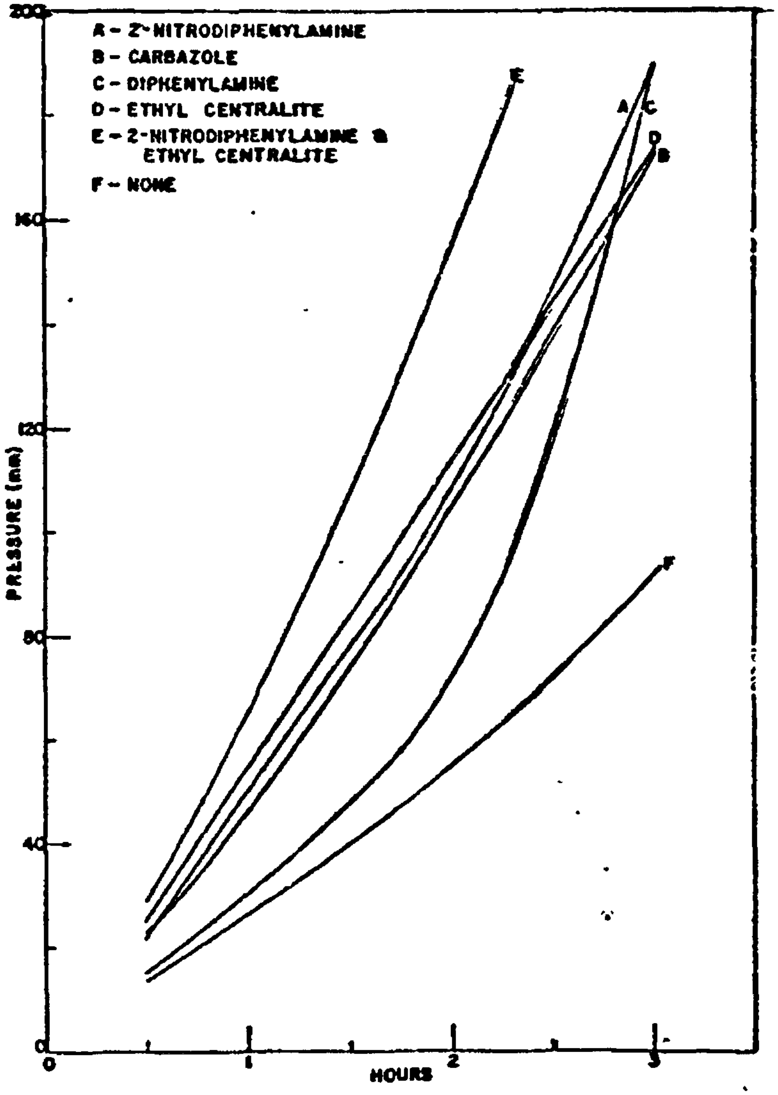

FIGURE 17. 110°C TALIANI TEST UNDER NITROGEN ON MODIFIED N-4 FORMULATIONS.

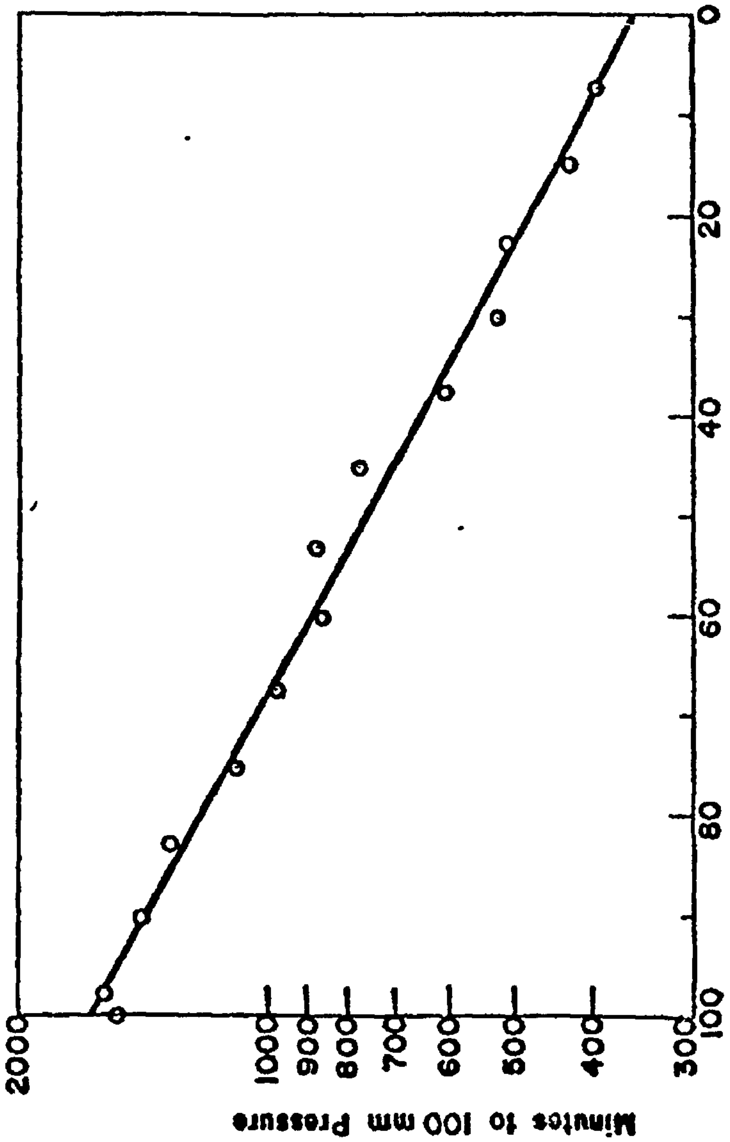

FIGURE 18. DETERMINATION OF RESIDUAL SAFE-LIFE OF N-4 STORED AT 80°C BY 110°C OXYGEN TALIANI TEST.

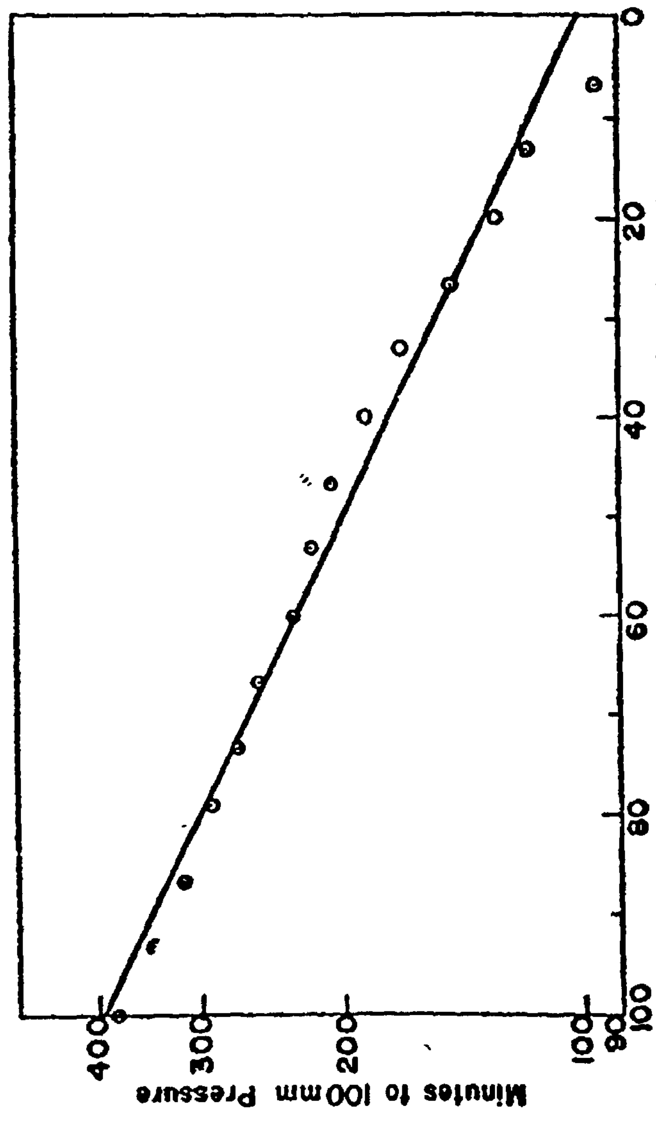

FIGURE 19. DETERMINATION OF RESIDUAL SAFE-LIFE OF M-4 STORED AT 80°C BY 120°C OXYGEN TALIANI TEST.

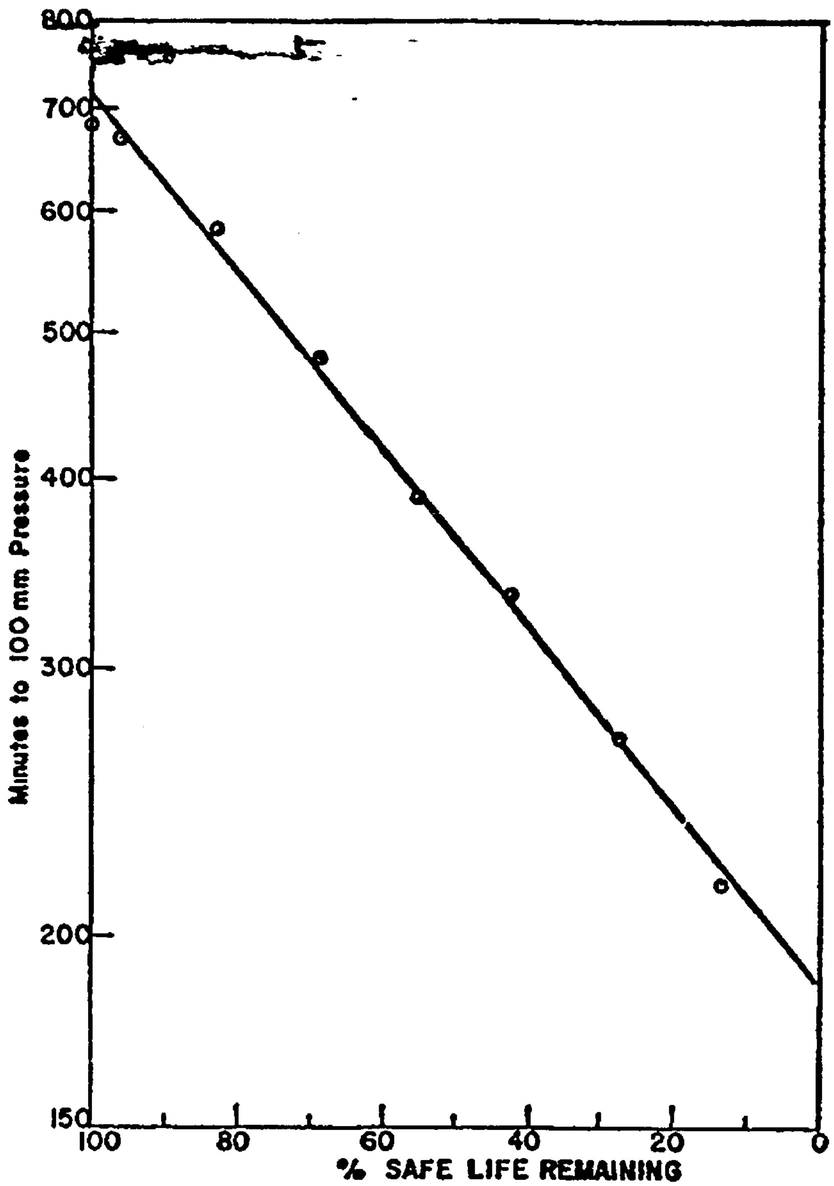

FIGURE 20. DETERMINATION OF RESIDUAL SAFE-LIFE OF JPN STORED AT 80°C BY 110°C OXYGEN TALIANI TEST.

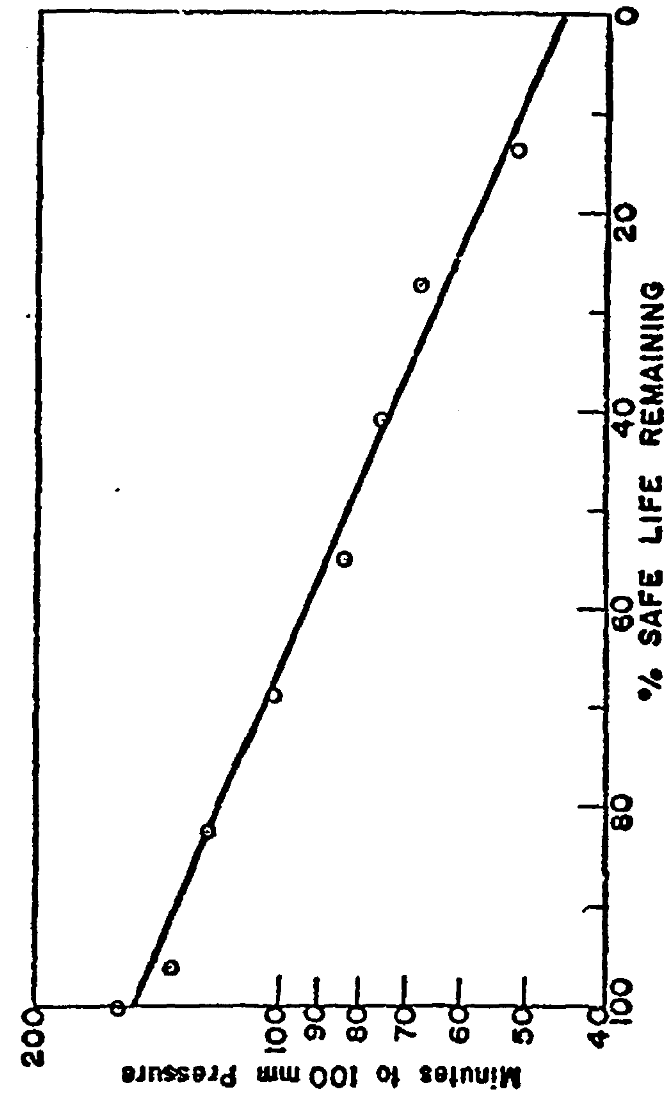

FIGURE 21. DETERMINATION OF RESIDUAL SAFE-LIFE OF JPN STORED AT 80°C BY 120°C OXYGEN TALIANI TEST.

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